- (1) Manganese; Mn; [7439-96-5]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION:

A number of authors have reported on the solubility of manganese in mercury near room temperature. The recommended solubility of 4.4×10^{-3} at % at 298 K was reported in two separate works by Krasnova and Zebreva (1) and Hurlen and Smaaberg (2); both groups employed potentiometry. Three other results support the recommended value: 3.6×10^{-3} at % at 293 K by Irvin and Russell (3); 4.6×10^{-3} at % at 293 K by Kemula and Galus (4); and 5.2×10^{-3} at % at 303 K by deWet and Haul (5). Chemical analysis (3,5) and voltammetry (4) were employed in the latter determinations.

There are several higher results but there is no basis for rejection of these data. Royce and Kahlenberg (6) determined a solubility of 1.13 x 10^{-2} at % at 293 K by chemical analysis. Jangg and Kirchmayr (7) reported a value of 6.8 x 10^{-3} at % at 288 K from potentiometric measurements. The solubility of 6.2 x 10^{-3} at % at 293 K reported by Ettmayer and Jangg (8) is slightly higher than the recommended value. Dowgird and Galus (9) employed potentiometry and determined the solubility to be 1.28 x 10^{-2} at % at 298 K. Sagadieva and Kozlovskii (10) used polarography to determine the solubility of 9.6 x 10^{-3} at % at 290 K.

Kozin (11) predicted a low solubility of 6.5×10^{-4} at % at 298 K. Some of the reported determinations of the solubility are rejected because the values were clearly too high, probably due to incomplete filtration: 0.014 (12), 0.44, 0.47 and 0.56 (13) at % at 282, 303, 328 and 343 K, respectively. The value of 9.2×10^{-4} at % reported by (14) is too low, probably because of corrosion of the manganese. Strachan and Harris (15) could not detect any dissolution of manganese in mercury at room temperature where their detection limit was 7×10^{-3} at %. Hickling and Maxwell (16) reported the solubility of 1.1×10^{-2} at % at 293 K but the work is not compiled due to lack of experimental details.

Jangg and Palman (17) determined the solubilities at 358 to 833 K, while Lange and coworkers (18,19,20) determined the solubilities over a temperature range of 293 to 368 K. There was agreement between (17) and (19) only in the region of 358 K. It may be that the dissolution of solid during electro-oxidation of the homogeneous amalgam resulted in increased estimates of the solubilities in (18-20). This system needs further investigation, especially in the temperature range of 300-600 K.

At temperatures below 345 \pm 3 K the liquid phase is reported to be in equilibrium with Mn2Hg5, while at 345 to 538 K the liquid is in equilibrium with MnHg (5,6,19-23).

Recommended (r) and tentative values of the solubility of Mn in Hg:

<u>T/K</u>	Soly/at %	Reference
293	3.6×10^{-3}	[3]
298	$4.5 \times 10^{-3} (r)^{a}$	[1,2,4]
357	8×10^{-2}	[17,19]
473	0.4	[17]
573	1.3	[17]
673	3	[17]
773	6	[17]

^aMean value from cited references.

(Continued next page)

- (1) Manganese; Mn; [7439-96-5]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION: (Continued)

References

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 deWet, J.F.; Haul, R.A.W. Z. Anorg. Chem. 1954, 277, 96.
 Royce, H.D.; Kahlenberg, L. Trans. Electrochem. Soc. 1931, 59, 126.
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 Dowgird, A.; Galus, Z. J. Electroanal. Chem. 1972, 34, 457.
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- 18. Lange, A.A.; Bukhman, S.P. Izv. Akad. Nauk Kaz. SSR, Ser. Khim. 1964, No. 3, 27. 19. Lange, A.A.; Bukhman, S.P.; Kozlovskii, M.T. Tr. Inst. Khim. Nauk Akad. Nauk Kaz. SSR 1969, 21, 92.
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- 22. Jangg, G.; Steppan, F. Z. Metallk. 1965, 56, 172.
- 23. deWet, J.F. Angew. Chem. 1955, 67, 208.

- (1) Manganese; Mn; [7439-96-5]
- (2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

Royce, H.D.; Kahlenberg, L.

Trans. Electrochem. Soc. 1931, 59, 121-33.

VARIABLES:

KIABLES:

One temperature: 20°C

PREPARED BY:

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The 20°C solubility of manganese in mercury was reported to be $(3.1 \pm 0.1) \times 10^{-3}$ mass %. The corresponding atomic % solubility calculated by the compilers is 1.1 x 10^{-2} at %. The liquid phase was reported to be in equilibrium with solid Mn₂Hg₅ up to 86°C. In the region of 86 to 100°C the solid phase was reported to be MnHg.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Heterogeneous amalgam was prepared electrolytically, then filtered through chamois skin. The liquid amalgam was analyzed by two methods: 1. A weighed sample of the amalgam was heated in conc. HCl for several hours to dissolve the Mn. Mercury was then washed, dried and weighed. 2. The Mn which was dissolved in HCl, as in the first method, was determined by the Volhard method.

SOURCE AND PURITY OF MATERIALS:

"Purest obtainable" materials were employed.

ESTIMATED ERROR:

Soly: accuracy ± 3%. Temp: not specified.

88 Manganese	
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Manganese; Mn; [7439-96-5]	Irvin, N.M.; Russell, A.S.
(2) Mercury; Hg; [7439-97-6]	J. Chem. Soc. 1932, 891-8.
VARIABLES:	PREPARED BY:
One temperature: 293 K	C. Guminski; Z. Galus
EXPERIMENTAL VALUES: The solubility of manganese in mercu The corresponding atomic % solubility	ry at 293 K was reported to be 1.0 \times 10 ⁻³ mass %. y calculated by the compilers is 3.6 \times 10 ⁻³ at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The heterogeneous amalgam was prepared by electrolysis. After equilibration the amalgam was filtered through a ground-glass filter. The separated liquid amalgam was shaken with acidified ferric sulfate to oxidize the Mn. Mercuric ions were then reduced by treatment with zinc amalgam, and manganese was determined volumetrically as permanganate after oxidation with sodium bismuthate and nitric acid.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: accuracy <u>+</u> 10%. Temp: not specified.

- (1) Manganese; Mn; [7439-96-5]
- (2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

deWet, J.F.; Haul, R.A.W.

Z. Anorg. Chem. 1954, 277, 96-112.

VARIABLES:

One temperature: 303 K

PREPARED BY:

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The 303 K solubility of manganese in mercury was reported to be 1.7 x 10^{-3} mass %. The corresponding atomic % solubility calculated by the compilers is 6.2 x 10^{-3} at %. The intermetallic compound, MnHg₄, was found to be in equilibrium with the homogeneous amalgam.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Amalgam was obtained by electrolysis of Mn(II)-sulfate at the mercury cathode and by the rotation of Mn rod in mercury at 30°C in a hydrogen atmosphere. The electrochemically obtained amalgam was filtered through sintered glass in a centrifuge vessel and sealed off under vacuum. After equilibration and sufficiently long centrifuging, about 10-12 grams of homogeneous amalgam was taken for analysis. This amalgam was treated with dilute phosphoric and sulfuric acids and the dissolved Mn, after oxidation with potassium periodate, was colorimetrically determined.

SOURCE AND PURITY OF MATERIALS:

 ${\rm MnSO_4\cdot 4H_2O}$ was Hopkins and Williams Analar grade.

Purified Hg was distilled under vacuum and was found spectrochemically free of Mn. All other chemicals and vessels used were carefully cleaned.

ESTIMATED ERROR:

Soly: accuracy approximately + 10%.

Temp: nothing specified.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6]	Kemula, W.; Galus, Z. Roczniki Chem. 1962, 36, 1223-38.
VARIABLES: One temperature: 20°C	PREPARED BY: C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of manganese in mercury at 20°C was found to be 3.1 x 10^{-3} mol dm³. The corresponding atomic % solubility calculated by the compilers is 4.6×10^{-3} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The heterogeneous manganese amalgam was prepared by electroreduction of Mn(II) on the hanging mercury-drop electrode. Then the peak of oxidation of the homogeneous amalgam was recorded under voltammetric conditions. The solubility was determined from the charge corresponding to this current peak and the volume of the mercury-drop.

SOURCE AND PURITY OF MATERIALS:

Analytically pure chemicals and doubly distilled water were used in the study.

ESTIMATED ERROR:

Soly: nothing specified; precision no higher than \pm 10% (compilers).

Temp: not specified.

COMPONENTS: (1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6] VARIABLES: ORIGINAL MEASUREMENTS: Jangg, G.; Palman, H. Z. Metallk. 1963, 54, 364-69.

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

Temperature: 86-565°C

The mass % solubility of manganese in mercury was presented graphically as a function of temperature. The data points were read from the curve and the solubilities were converted to atomic % by the compilers.

t/°C	Soly/at %	<u>t/°C</u>	Soly/at %
86	0.087	300	1.3
100	0.10	330	1.9
114	0.12	350	2.2
125	0.17	370	2.6
148	0.26	400	3.1
166	0.31	418	3.6
198	0.36	450	4.6
225	0.51	470	5.6
246	0.69	500	6.3
270	0.87	552	7.6
		565	8.2

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Amalgam preparation was not specified. At below 320°C the amalgam was equilibrated for 12 hr in a glass vessel, after which the amalgam was filtered and analyzed. Above 320°C the heterogeneous amalgam was introduced into specially constructed apparatus made of refractory Cr-steel. apparatus could be used because of very low solubility of Fe in Hg, and because the Cr(III)-oxide film inhibits the wetting of the steel by Hg. After 12 hr of equilibration at the temperature of the experiment, the amalgam was filtered through the sintered iron frit under a pressure of purified nitrogen. Usually 3- to 4-fold filtration was necessary. The metal content was then analytically determined in the filtered saturated amalgam. Analytical procedure not described.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: precision better than \pm 5%.

Temp: precision ± 2 K.

COMPONENTS: (1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6] VARIABLES: One temperature: 15°C ORIGINAL MEASUREMENTS: Jangg, G.; Kirchmayr, H. Z. Chem. 1963, 3, 47-56.

EXPERIMENTAL VALUES:

The data were reported graphically; a solubility of 4.6 x 10^{-3} mol dm⁻³ at 15°C was read from the curve by the compilers. The corresponding atomic % solubility calculated by the compilers is 6.8 x 10^{-3} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The amalgams were obtained by electrolysis with 100% efficiency. Concentration of the amalgam was determined by coulometry. Potentials of the Mn-amalgam were measured against the SCE in the cell,

 $\mathrm{Mn(Hg)}_{\mathrm{x}} | \mathrm{0.01-1.0} \ \mathrm{mol} \ \mathrm{dm}^{-3} \ \mathrm{MnSO}_{4} | \mathrm{KCl}$, $\mathrm{Hg}_{2}\mathrm{Cl}_{2}$, Hg

The concentration of the saturated amalgam was evaluated from the breakpoint in the plot of the potential vs. logarithm of the amalgam concentration. The experiments were performed in an atmosphere of nitrogen.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: error may be as high as \pm 50%. Temp: precision better than \pm 1 K.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6]	Sagadieva, K.Zh.; Kozlovskii, M.T. Vestn. Akad. Nauk Kaz. SSR 1963, No. 5, 85-7.
VARIABLES:	PREPARED BY:
One temperature: 17°C	C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of manganese in mercury at 17°C was found to be $(6.5 \pm 0.1) \times 10^{-3}$ mol dm⁻³. The corresponding atomic % solubility calculated by the compilers is 9.6 x 10^{-3} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The amalgam was prepared by electrolytic deposition of Mn on Hg cathode with 100% current efficiency. The concentration of the amalgam was determined from the current and time of the electrolysis. The solubility was determined polarographically; the anodic current was practically independent of the Mn content when amalgam saturation was attained. Oxidation of the Mn amalgam was carried out in two background electrolyte: 0.1 mol dm $^{-3}$ KNO3 and 1 mol dm $^{-3}$ ammonia buffer.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: error probably \pm 3% (compilers).

Temp: precision ± 1 K.

COMPONENTS: (1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6] VARIABLES: Temperature: 20-50°C CRIGINAL MEASUREMENTS: Lange, A.A.; Bukhman, S.P. Izv. Akad. Nauk Kaz. SSR, Ser. Khim. 1964, No. 3, 27-32.

EXPERIMENTAL VALUES:

The mass % solubility of manganese in mercury was reported graphically; only the numerical value for 20°C was presented by the authors. The solubility at 30, 40, and 50°C was read from the curve by the compilers, and the corresponding atomic % solubilities were calculated for all temperatures.

<u>t/°C</u>	Soly/mass $\% \times 10^3$	Soly/at $\% \times 10^3$
20	3.42	12.5
30	6.7	24
40	11	40
50	13	48

These results were also presented in (1).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared electrolytically. The amalgams were oxidized under voltammetric conditions and current-potential curves were constructed. For amalgams with manganese content exceeding its solubility in mercury the limiting current was constant, while in the region of homogeneity the current was linearly dependent on concentration. The concentration of the amalgam corresponding to the change of the character of this dependence was taken as the concentration equal to the solubility of manganese in mercury.

SOURCE AND PURITY OF MATERIALS:

Not specified.

ESTIMATED ERROR:

Soly: not specified; error probably less

than \pm 10% (compilers).

Temp: nothing specified.

REFERENCES:

 Lange, A.A.; Bukhman, S.P. Elektrokhimia 1969, 5, 553.

COMPONENTS: (1) Manganese, Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6] VARIABLES: One temperature: 25°C ORIGINAL MEASUREMENTS: Krasnova, I.E.; Zebreva, A.I. Elektrokhimia 1966, 2, 96-9. PREPARED BY: C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of manganese in mercury at 25°C was reported to be 1.2×10^{-3} mass %. The corresponding atomic % solubility calculated by the compilers is 4.4×10^{-3} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The manganese amalgams of various concentrations were prepared by the electroreduction of manganese (II) on the hanging mercury drop electrode. The oxidation current of manganese from these amalgams was then recorded under voltammetric conditions. By plotting the peak current value versus the amalgam concentration, the change of the character of this dependence at the saturation point was observed. This enabled the determination of the solubility.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: error \pm 33%. Temp: precision \pm 2 K.

- (1) Manganese; Mn; [7439-96-5]
- (2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

Lange, A.A.; Bukhman, S.P.; Kozlovski, M.T. Tr. Inst. Khim. Nauk, Akad. Nauk Kaz. SSR

1969, 21, 92-102.

VARIABLES:

PREPARED BY:

Temperature: 20-95°C

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

Solubility of manganese in mercury:

t/°C	Soly/mol dm ⁻³	Soly/at % ^a
20	8.46×10^{-3}	0.012
30	1.68×10^{-2}	0.025
40	2.65×10^{-2}	0.039
50	3.23×10^{-2}	0.047
70	4.84×10^{-2}	0.071
80	5.6×10^{-2}	0.082
82	7.00×10^{-2}	0.098
85	7.60×10^{-2}	0.11
88	9.90×10^{-2}	0.14
90	1.11×10^{-1}	0.16
95	1.23×10^{-1}	0.18

^aby compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The amalgams were prepared by the electroreduction of Mn(II) on the Hg cathode.
The solubilities were determined on the
basis of the limiting current of the
manganese amalgam oxidation. When the
content of metal in mercury exceeded the
solubility the current ceased to be
dependent on the manganese content.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: nothing specified; error may be as high as 4% (compilers).

Temp: nothing specified.

- (1) Manganese; Mn; [7439-96-5]
- (2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

Dowgird, A.; Galus, Z.

J. Electroanal. Chem. 1972, 34, 457-61.

VARIABLES:

PREPARED BY:

One temperature: 25°C

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

Solubility of manganese in mercury at 25°C was reported to be 8.7 x 10^{-3} mol dm⁻³. The corresponding atomic % solubility calculated by the compilers is 1.28×10^{-2} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The amalgam was prepared by reduction of Mn(II) at constant current density on the hanging mercury—drop electrode. Then the potential was measured with respect to SCE over a period of 12 min. At higher concentrations the potential changes were only observed in 60 sec. The experiments were performed in hydrogen atmosphere. The inflection point of the plot of potential vs. logarithm of concentration corresponded to the saturation point. It is probable that the amalgams were slightly supersaturated at the highest concentrations.

SOURCE AND PURITY OF MATERIALS:

All chemicals were of reagent grade. Mercury was chemically purified by prolonged shaking with acidified solution of ${\rm Hg_2(NO_3)_2}$ then double-distilled at reduced pressure. All solutions prepared with triply-distilled water.

ESTIMATED ERROR:

Soly: nothing specified; precision no better than + 10% (compilers).

Temp: precision + 0.2 K.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6]	Ettmayer, P.; Jangg, G. Monatsh. Chem. 1973, 104, 1120-30.
VARIABLES:	PREPARED BY:
One temperature: 293 K	C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of manganese in mercury at 293 K was reported to be 1.7 x 10^{-3} mass %. The corresponding atomic % solubility calculated by the compilers is 6.2 x 10^{-3} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The amalgam was prepared by electroreduction of Mn(II) on a Hg cathode. The electrolyte contained MnSO₄ and (NH₄)₂SO₄ as a buffer. The amalgam was separated from the electrolyte, dried, filtered, and analyzed by an unknown method.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: nothing specified; accuracy probably no better than \pm 10% (compilers).

Temp: nothing specified.

COMPONENTS: (1) Manganese; Mn; [7439-96-5] (2) Mercury; Hg; [7439-97-6] VARIABLES: One temperature: 25°C ORIGINAL MEASUREMENTS: Hurlen, T.; Smaaberg, R. J. Electroanal. Chem. 1976, 71, 157-68.

EXPERIMENTAL VALUES:

The solubility of manganese in mercury at 25°C was reported to be 1.2 x 10^{-3} mass %. The corresponding atomic % solubility calculated by the compilers is 4.4 x 10^{-3} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Amalgams of various concentrations were prepared electrolytically. The potentials of such amalgams, in the presence of a constant Mn(II) concentration, were measured in the cell,

measured in the cell, $Mn(Hg)_x|n mol dm^{-3} MnCl_2$, (0.5-n) mol dm⁻³ $MgCl_2$, pH = 4.3-4.9 | KCl, AgCl, Ag

Up to the saturation point the potential of the amalgam was dependent on its concentration; at higher concentrations the potential was constant. The solubility was determined from the inflection point of the potential-concentration dependence.

SOURCE AND PURITY OF MATERIALS:

Analytically pure reagents and doubly-distilled water were used.

ESTIMATED ERROR:

Soly: nothing specified; precision better than \pm 10% (compilers).

Temp: not specified.

300 Rhenium

COMPONENTS: **EVALUATOR:** (1) Rhenium; Re; [7440-15-5] C. Guminski; Z. Galus Department of Chemistry (2) Mercury; Hg; [7439-97-6] University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

No specified data on the solubility of rhenium in mercury has been published. It has been reported (1) that rhenium powder is not attacked by mercury when the metals are heated in a reducing atmosphere at 573 K. Also, Jangg and Dörtbudak (2) equilibrated the two metals at 773 K and could not detect any dissolution of rhenium; their analytical detection limit was 10^{-5} at %. Kozin (3) estimated that the solubility of rhenium in mercury at 298 K is 5.9 x 10^{-18} at %; a previously predicted value of 3.5 x 10^{-29} at % at 298 K appears to be less probable (4).

The solid phase in equilibrium with the saturated rhenium amalgam should contain pure rhenium because no Re-Hg compound was found (2).

References

- 1. Heyne, R.; Moers, K. Z. Anorg. Chem. 1931, 196, 151.

- Jangg, G.; Dörtbudak, T. Z. Metallk. 1973, 64, 715.
 Kozin, L.F. Tr. Inst. Khim. Nauk Akad. Nauk Kaz. SSR 1962, 9, 101.
 Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii, Nauka, Alma-Ata, 1964.